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L6: Entry 5 of 16

File: USPT

May 15, 1990

DOCUMENT-IDENTIFIER: US 4925503 A

TITLE: Solid explosive and propellant compositions containing a polyurethane polyacetal elastomer binder and method for the preparation thereof

BSPR:

Recent technical papers on LOVA binders were presented at the "ONR Workshop on Energetic Polymer and Processing Science" given in Chestertown, Md. on July 25-27, 1983 and reproduced in CPIA Publication 405, Chemical Propulsion Information Agency (June, 1984). In a paper reproduced on pages 131-139 of CPIA Publication 405, J. C. W. Chien generally describes the "Synthesis of Polyacetal-Polyurethane Thermoplastic Elastomers as LOVA Binders" by end capping methylene(bisphenylisocyanate (MDI) or toluene diisocyanate (TDI) to form a prepolymer, and then chain extending this prepolymer with a monomer which is either bis(2-hydroxyethyl) terephthalamide or bis(2-hydroxyethyl-isophthalamide). The testing of these types of polyurethane polyacetals formed by reacting an isocyanate-terminated prepolymer with a monomer is disclosed in an article by S. T. Peters et al entitled "Characterization of Thermoplastic Elastomers for Use in Gun Propellants" from Proceedings of the American Defense Preparedness Association; Chemical & Plastics Section of the Chemical Systems Division, held October 27-29, 1986, Long Beach, Calif., pp. 8-15. Table I at page 12 of this article discloses, in Samples 17 through 22 of the table, physical property data on these types of polymers. These samples show thermal stability, when loaded with 50 percent of RDX in a binder/propellant formulation, up to a decomposition temperature peak of between 191.degree. C. and 210.degree. C., as compared to the decomposition temperature of RDX alone of 220.degree. C. This difference in decomposition temperature between the pure RDX and the RDX/binder mixtures indicates some interaction between RDX and these binders, believed to be an undesirable effect.

BSPR:

The polyacetal homopolymer soft segment of the elastomer can be prepared by known reactions. In accordance with a preferred reaction, a 4-lower alkyl substituted-1,3-dioxolane (preferably 4-methyl-1,3-dioxolane) is reacted with boron trifluoride etherate at a reaction temperature of between about -10.degree. C. and about -20.degree. C., preferably between about -15.degree. C. and about -20.degree. C., for a reaction time of at least about 8 hours, preferably at least 16 hours, although the reaction can take as long as 70 hours or longer depending upon the specific reactants and reactor conditions. The reaction is then quenched, and the resulting product is washed, hydrolyzed, and dried to provide the desired polyacetal homopolymer. This type of reaction is described in further detail in a technical article by M. Okada et al entitled "Polymerizability of Methyl Substituted 1,3-Dioxolanes" appearing in Die Makromolekulare Chemie, Vol. 176, pp. 859-872 (1975), incorporated herein by reference. The hydrolysis step serves to cleave any hemiacetal terminal groups and replace them with alcohol groups (which are more reactive). The desired homopolymer generally has a weight average molecular weight of between about 1,000 and about 60,000 (preferably between about 5,000 and about 40,000, more preferably between about 10,000 and about 40,000) as measured by gel permeation chromatography against a polystyrene standard.

BSPR:

The polyacetal copolymer useful in the present invention is the reaction product of 1,3-dioxolane with a formaldehyde-providing compound. Representative formaldehyde-providing compounds include, for example, trioxane, formaldehyde, paraformaldehyde, and combinations thereof. This reaction is generally conducted at between about 20.degree. C. and about 70.degree. C., preferably between about

25.degree. C. and about 50.degree. C., for up to a day or more, preferably between about 3 and about 24 hours to provide the desired copolymer. This reaction is described in more detail in a technical article by M. Inoue entitled "Crystallization and Melting of Copolymers" appearing in Journal of Applied Polymer Science, Vol. 8, pp. 2225-2238 (1964), incorporated herein by reference. The desired copolymer generally has a weight average molecular weight of between about 1,000 and about 200,000 (preferably between about 5,000 and about 150,000, more preferably between about 10,000 and about 100,000) as measured by gel permeation chromatography against a polystyrene standard.

BSPV:

(a) reacting a dihydroxy-terminated polyacetal homopolymer with an alkylene diisocyanate in the presence of a urethane forming catalyst in a reaction to produce an isocyanate-terminated prepolymer, said homopolymer having a weight average molecular weight of between about 1,000 and about 60,000 as measured against a polystyrene standard by gel permeation chromatography and being produced by polymerizing a 4-lower alkyl substituted-1,3-dioxolane, said reaction employing between about 50 mole percent and about 70 mole percent of said alkylene diisocyanate based upon the total amount of said alkylene diisocyanate and said homopolymer employed,

BSPV:

(b) reacting said isocyanate-terminated prepolymer with a dihydroxy-terminated polyacetal copolymer in the presence of a solvent and a urethane-forming catalyst to provide a polyurethane polyacetal elastomer, said copolymer having a weight average molecular weight of between about 1,000 and about 200,000 as measured against a polystyrene standard by gel permeation chromatography and being the reaction product of a formaldehyde-providing compound and 1,3-dioxolane, said copolymer being employed in an amount of between about 30 and about 50 mole percent based upon the total amount of said copolymer and said isocyanate-terminated prepolymer employed, and

BSPV:

(a) reacting a dihydroxy-terminated polyacetal copolymer with an alkylene diisocyanate in the presence of a urethane-forming catalyst in a reaction to produce an isocyanate-terminated prepolymer, said copolymer having a weight average molecular weight of between about 1,000 and about 200,000 as measured against a polystyrene standard by gel permeation chromatography and being the reaction product of a formaldehyde-providing compound and 1,3-dioxolane, said reaction employing between about 50 mole percent and about 70 mole percent of said alkylene diisocyanate based upon the total amount of said alkylene diisocyanate and said copolymer employed,

BSPV:

(b) reacting said isocyanate-terminated prepolymer with a dihydroxy-terminated polyacetal homopolymer in the presence of a solvent and a urethane-forming catalyst to provide a polyurethane polyacetal elastomer, said homopolymer having a weight average molecular weight of between about 1,000 and about 60,000, as measured against a polystyrene standard by gel permeation chromatography and being produced by polymerizing a 4-lower alkyl substituted-1,3-dioxolane, said homopolymer being employed in an amount of between about 30 and about 50 mole percent based upon the total amount of said homopolymer and said isocyanate-terminated prepolymer employed, and

BSPV:

(a) reacting a dihydroxy-terminated polyacetal homopolymer with an alkylene diisocyanate in the presence of a urethane forming catalyst in a reaction to produce an isocyanate-terminated prepolymer, said homopolymer having a weight average molecular weight of between about 1,000 and about 60,000 as measured against a polystyrene standard by gel permeation chromatography and being produced by polymerizing a 4-lower alkyl substituted-1,3-dioxolane, said reaction employing between about 50 mole percent and about 70 mole percent of said alkylene diisocyanate based upon the total amount of said alkylene diisocyanate and said homopolymer employed, and

BSPV:

(b) reacting said isocyanate-terminated prepolymer with a dihydroxy-terminated polyacetal copolymer in the presence of a solvent and a urethane forming catalyst to provide said elastomer, said copolymer having a weight average molecular

weight of between about 1,000 and about 200,000 as measured against a polystyrene standard by gel permeation chromatography and being the reaction product of a formaldehyde

DEPR:

The polymer was characterized via Gel Permeation Chromatography, which showed it had a molecular weight of 35,291 versus a polystyrene standard (using a linear ULTRASTRYAGEL column and chloroform as a solvent).

DEPR:

A portion of this was fractionated from methylene chloride to give about an 80 percent recovery of polymer. Gel Permeation Chromatography showed a molecular weight of 138,701 versus a polystyrene standard (using a linear ULTRASTRYAGEL column and chloroform as a solvent).

DEPR:

After 10.8 hours this solution was poured into a previously made solution of 18.5 g (0.0049 mole) trioxane/1,3-dioxolane co-polymer and 37.2 g of 1,1,2,2-tetrachloroethane (at 65.degree. C.). The combined solutions were stirred at 65.degree. C. for two days, then 0.10 g (0.00016 mole) dibutyltin dilaurate was added and the reaction was continued for an additional day. Petroleum ether was added to the reaction mixture and the solvent layer decanted away from the polymer. After washing with more petroleum ether, the process was repeated and the polymer dried at 50.degree. C. under vacuum (0.1 mm) several days. A yield of 57.6 g was obtained. Gel Permeation Chromatography of the polymer showed a weight average molecular weight of 166,783 versus a polystyrene ; standard using a linear ULTRASTRYAGEL column and chloroform as a solvent. Note that this molecular weight is a weight average using a specific (polystyrene) GPC standard and cannot necessarily be calculated by merely adding the GPC standard molecular weights of the component segments comprising the elastomer. The NMR (proton), run in deuterated chloroform, indicated between three and four soft segments per hard segment. Differential Scanning Calorimetry (DSC) showed a glass transition point about -45.degree. C. and a melting point at 86.5.degree. C. (run under inert atmosphere). This melting point is considered ideally suited for extrusion melt processing in contrast to typical prior art polyurethane polyacetal binders having much higher melting points.

CLPV:

(a) reacting a dihydroxy-terminated polyacetal homopolymer with an alkylene diisocyanate in the presence of a urethane-forming catalyst in a reaction to produce an isocyanate-terminated prepolymer, said homopolymer having a weight average molecular weight of between about 1,000 and about 60,000 as measured against a polystyrene standard by gel permeation chromatography and being produced by polymerizing a 4-lower alkyl substituted-1,3-dioxolane, said reaction employing between about 50 mole percent and about 70 mole percent of said alkylene diisocyanate based upon the total amount of said alkylene diisocyanate and said homopolymer employed,

CLPV:

(b) reacting said isocyanate-terminated prepolymer with a dihydroxy-terminated polyacetal copolymer in the presence of a solvent and a urethane forming catalyst to provide a polyurethane polyacetal elastomer, said copolymer having a weight average molecular weight of between about 1,000 and about 200,000 as measured against a polystyrene standard by gel permeation chromatography and being the reaction product of a formaldehyde-providing compound and 1,3-dioxolane, said copolymer being employed in an amount of between about 30 and about 50 mole percent based upon the total amount of said copolymer and said isocyanate-terminated prepolymer employed, and

CLPV:

(a) reacting a dihydroxy-terminated polyacetal copolymer with an alkylene diisocyanate in the presence of a urethane-forming catalyst in a reaction to produce an isocyanate-terminated prepolymer, said copolymer having a weight average molecular weight of between about 1,000 and about 200,000 as measured against a polystyrene standard by gel permeation chromatography and being the reaction product of a formaldehyde-providing compound and 1,3-dioxolane, said, reaction employing between about 50 mole percent and about 70 mole percent of said alkylene diisocyanate based upon the total amount of said alkylene diisocyanate and said copolymer employed, and

CLPV:

(b) reacting said isocyanate-terminated prepolymer with a dihydroxy-terminated polyacetal homopolymer in the presence of a solvent and a urethaneforming catalyst to provide said elastomer, said homopolymer having a weight average molecular weight of between about 1,000 and about 60,000, as measured against a polystyrene standard by gel permeation chromatography and being produced by polymerizing a 4-lower alkyl substituted-1,3-dioxolane, said homopolymer being employed in an amount of between about 30 and about 50 mole percent based upon the total amount of said homopolymer and said isocyanateterminated prepolymer employed, and

CCOR:

149/19.4

CCXR:

149/19.6

ORPL:

J. C. W. Chien, "Synthesis of Polyacetal-Polyurethane Thermoplastic Elastomers as LOVA-Binders", University of Massachusetts, Amherst, MA, ONR WORKSHOP ON ENERGETIC POLYMER AND PROCESSING SCIENCE, CPIA Publication 405, Jun. 1984, Jul. 25-27, 1983, pp. 131-139.

ORPL:

Baobei Xu, D. N. Khanna, C. Peter Lillya, and James C. W. Chien, "Structure-Property Relationships in Thermoplastic Elastomers III. Segmented Polyacetal-Polyurethanes", JOURNAL OF APPLIED POLYMER SCIENCE, vol. 31, pp. 123-133, (1986).

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L6: Entry 8 of 16

File: USPT

Nov 26, 1985

DOCUMENT-IDENTIFIER: US 4555277 A

TITLE: Extrusion cast explosive

BSPR:

U.S. Pat. No. 3,778,319 to Benzinger et al., describes a high energy plastic-bonded explosive of 95% HMX, and 2.5% DNPA/F which is a eutectic mixture of the formal and acetal of 2,2-dinitropropanol as the plasticizer. The plasticizer is a thermoplastic, polyurethane elastomer, manufactured by B. F. Goodrich Company and sold under the trade name of Estane, which is prepared from long chain diols and toluene diisocyanate.

DEPR:

PVF was obtained from Monsanto Chemical Company and is sold under the trade name of Formvar. In all the compositions listed below, PVF 5/95 was used. PVF 5/95 is polyvinylformal, having a molecular weight of approximately 12,000, $f=15$, equivalent weight of 830 and a density of 1.227 g/cc. PCL 240, with a molecular weight of about 1986, $f=2.0$, equivalent weight of 993, and a density of 1.07 g/cc, was used in the compositions, and was obtained from Union Carbide Corporation.

CCOR:

149/19.4

CCXR:

149/88

CCXR:

149/92

WEST**End of Result Set**

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L6: Entry 16 of 16

File: USPT

Oct 10, 1972

DOCUMENT-IDENTIFIER: US 3697668 A
TITLE: SELF-DESTRUCTIBLE MOLDED ARTICLES

DEPR:

In order to obtain the full measure of benefits afforded by this invention the total concentration of matrix resin (excluding capsule wall material) and fibrous reinforcement must be within a certain range compared to the concentration of matrix-confined encapsulated incendiary material. Moreover, the concentration of fibrous reinforcement versus matrix resin must also be controlled within a certain range in cases where fibrous materials are used which are resistant to burning, e.g., glass fibers, metal fibers, etc. Also the concentration of encapsulated incendiary material versus capsule cell wall material should be within fixed limits to insure adequate burning yet preserve the inertness of the incendiary material until the desired time for thermal destruction. Of the total core section (including fibrous-reinforced resin matrix plus encapsulated incendiary material, viz., everything but printed circuitry, facing sheet(s), igniting mechanisms, and electronic component(s) structure), the fibrous-reinforced resin matrix should constitute from about 15 to 45 weight percent and the encapsulated incendiary (including capsule cell wall material) should constitute from about 55 to 85 weight percent. When the fibrous reinforcement is a material resistant to burning, e.g., glass fibers; the fibrous reinforcement should constitute from 10 to 35 weight percent of the total of resin matrix plus fibrous reinforcement with the remainder being readily burnable matrix resin. When the fibrous reinforcement is a readily burnable material (at the combustion temperatures produced by the encapsulated incendiary); the matrix resin should constitute a major portion of the total of matrix resin plus fibrous reinforcement as in such cases the burnable fibrous reinforcement material is usually a higher molecular weight plastic or resin or one having a higher melting temperature (to preserve its fibrous integrity during molding and its reinforcing power thereafter) and such materials usually require the expenditure of more thermal energy to attain substantially complete combustion thereof than is required for combustion of the non-fibrous matrix resin.

DEPR:

Both thermoplastic and thermosetting resins can be used, e.g., in liquid or powdered form, as readily combustible matrix resins. Suitable exemplary resins include, but are not limited to the following: cellulosic resins, e.g., cellulose acetate, cellulose butyrate, cellulose nitrate, nitrocellulose; polyolefins, e.g., polyethylene, polypropylene, polybutenes, ethylene-propylene copolymers including terpolymers of ethylene, propylene and a C.sub.4 to C.sub.8 conjugated or non-conjugated diolefin, polybutadienes; polyesters, e.g., reaction products of polyhydric alcohols, such as ethylene glycol, diethylene glycol, glycerol, pentaerythritol, trimethylol propane, polybutylene glycols, castor oil, etc., with polybasic organic acids, e.g., oxalic, adipic, sebacic, phthalic, isophthalic, terephthalic, 6-amino caproic, malonic, succinic, maleic, fumaric, itaconic, etc.; polyurethanes, e.g., those prepared by reacting polyhydric materials, including those mentioned above, as well as hydroxy containing polyethers, polyesters, polyamines, etc., with polyisocyanates, usually aryl diisocyanates, e.g., toluene diisocyanate, or triisocyanates; epoxy resins, e.g., reaction products of epihalohydrins, such as epichlorohydrin, with bisphenols, such as "Bisphenol-A" (p, p'-isopropylidene diphenol). Mixtures of any two or more of the above or equivalent resins can likewise be employed.

DEPR:

Two separate ethylene-maleis anhydride (EMA) copolymer aqueous solutions are

prepared with each having a two percent by weight concentration of the respective EMA material and the pH of each solution being adjusted to 9 using 10 percent aqueous sodium hydroxide. One EMA solution is formed using an ethylene-maleic anhydride copolymer having a molecular weight of approximately 6,000, e.g., a commercially available Monsanto Company product designated "EMA-21." The other EMA solution is formed using a copolymer of ethylene and maleic anhydride having a molecular weight ranging from 60,000 to 70,000, e.g., a commercially available Monsanto Company product designated "EMA-31."

DEPR:

Then 200 grams of the internal phase material are dispersed in the external phase sols by charging 180 grams of the gelatin solution and 180 grams of the gum arabic solution (pH adjusted to 9) to a coacervation vessel, e.g., a three liter glass beaker, followed by the addition of 1,500 cubic centimeters of deionized water and 40 grams of the aqueous solution of the 6,000 molecular weight copolymer of ethylene and maleic anhydride. The resulting solution is heated to 45.degree. C. while stirring, e.g., using a flat-bladed impeller. The internal phase material is then added to the heated solution and stirring is continued to achieve proper dispersion.

DEPR:

After dispersion, the solution pH is lowered to 5 with 10 percent by weight acetic acid aqueous solution. Thin walls appear and coacervate spheres appear in the external phase. The pH is then raised to 6 with 20 weight percent sodium hydroxide aqueous solution to decoacervate the spheres. Then 40 grams of the aqueous solution of the 60,000 to 70,000 molecular weight ethylene-maleic anhydride copolymer is added and the pH is lowered to 5.3 with 10 percent by weight aqueous acetic acid. As the pH is lowered, capsule wall formation becomes thicker. Maximum thickness is obtained by cooling to approximately 25.degree. C. at pH of 5.3.

DEPR:

When encapsulating with a nitrocellulose external phase material, a phase separation inducing agent is usually employed, e.g., a commercially available, carboxy-terminated, low molecular weight, liquid polybutadiene homopolymer (molecular weight of approximately 2,500) marketed under the trade designation "Butarez." The following parameters are usually observed:

CCXR:

149/2

CCXR:

149/20

CCXR:

149/21

CCXR:

149/38

CCXR:

149/42

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l3 and (polyurethane or polyisocyanate or thermoplastic adj elastomer)	12

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Refine Search:

l3 and (polyurethane or polyisocyanate
or thermoplastic adj elastomer)

Clear**Search History****Today's Date: 11/4/2001**

<u>DB Name</u>	<u>Query</u>	<u>Hit Count</u>	<u>Set Name</u>
USPT,PGPB	l3 and (polyurethane or polyisocyanate or thermoplastic adj elastomer)	12	<u>L4</u>
USPT,PGPB	l2 and ((149/\$)!.CCLS.)	27	<u>L3</u>
USPT,PGPB	pentanediol or pentane adj diol	11170	<u>L2</u>
USPT,PGPB	pentanediol or pentand adj diol	9694	<u>L1</u>

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L4: Entry 2 of 12

File: USPT

Apr 21, 1998

DOCUMENT-IDENTIFIER: US 5741998 A

TITLE: Propellant formulations based on dinitramide salts and energetic binders

BSPR:

A currently preferred class of nitramine polymers are polyesters based on nitraminodiacetic acid and a diol or mixture of diols. Suitable diols are aliphatic diols containing from 2-6 carbon atoms and primary alcohol functional groups. The diol can contain ether linkages, but the diol molecule preferably does not also contain an ester or ketone. Examples of suitable diols include 1,2-ethanediol, 1,3-propanediol, 1,4-butanediol, 1,5-pentanediol, 1,6-hexanediol, diethyleneglycol, triethyleneglycol, tetraethyleneglycol, and 2,2-dinitro-1,3-propanediol.

DEPL:

The curatives and stabilizers included 0.4% NO.sub.x scavenger MNA (N-methyl-p-nitroaniline), 3.11% Desmodur.RTM. N-100, a polyisocyanate curative obtained from Mobay, 0.05% acid scavenger (N,N,N',N'-tetramethyl-1,8-naphthalenediamine, obtained from Aldrich), and 0.005% cure catalyst TPB (triphenyl bismuth).

CLPR:

20. A composite propellant formulation as defined in claim 19, wherein the diol is selected from 1,2-ethanediol, 1,3-propanediol, 1,4-butanediol, 1,5-pentanediol, 1,6-hexanediol, diethyleneglycol, triethyleneglycol, tetraethyleneglycol, 2,2-dinitro-1,3-propanediol, and mixtures thereof.

CCOR:

149/19.6

CCXR:

149/19.4

WEST

Generate Collection

L4: Entry 3 of 12

File: USPT

Aug 29, 1989

DOCUMENT-IDENTIFIER: US 4861397 A
TITLE: Fire-resistant explosives

ABPL:

A fire resistant composition comprising an explosive in an amount of 41-85%, an additive selected from the group consisting of zinc borate, hexabromobiphenyl, molybdenum flame suppressant, triaryl phosphate ester, calcium formate, antimony oxide, ammonium phosphate, aluminum oxide trihydrate, and organophosphorous diols in an amount of 9-41% and a binder component selected from the group consisting of polyurethane, acrylic polymers, phosphate ester-vinyl chloride latexes, cellulose acetate butyrate, vinyl esters, styrene-ethylene butylene block copolymers fluorinated elastomers, and Plaster of Paris rubberized with acrylic latexes in an amount of 6-39%, all of proportions being on a % by weight basis.

BSPR:

Specific operable binders include polyurethanes, acrylic polymers, phosphate ester-vinyl chloride latexes, cellulose acetate butyrate, vinyl esters, styrene-ethylene-butylene block copolymers, fluorinated elastomers, and Plaster of Paris rubberized with acrylic latexes. The amounts of binder ranged from about 6 to 39% by weight. Plasticizers may be added to improve processing or final products properties.

DEPR:

98 grams of a brominated vinyl ester resin (Dow Derakane 510N), 1.5 grams of methyl ethyl ketoneperoxide having 8.8% active oxygen (Lupersol DHD-9) 0.3 grams of a solution of cobalt naphthenate having 6% cobalt and 0.05 grams of dimethyl aniline were mixed together. To the resultant mixture there was added 300 grams of class 1 RDX and 100 grams of class 5 RDX with thorough stirring. In order to allow more time for mixing the ingredients, the methylethyl ketoneperoxide may be added later or materials such as, 2,4-pentane diol may be added to delay the curing process. A castable, rigid composition forms within 24 hours. A sample of the composition was subjected to the Hot Wire and Open Flame tests. In comparison with an untreated composition, ignition was delayed.

CCOR:

149/19.4

CCXR:

149/19.1

CCXR:

149/19.91

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L4: Entry 4 of 12

File: USPT

Feb 7, 1984

DOCUMENT-IDENTIFIER: US 4430131 A
TITLE: Polyurethane binders

BSPR:

This invention relates to a new class of polyesters and to novel polyurethane-type elastomer binders useful in solid propellants, formed by reacting the novel polyesters with a diisocyanate.

BSPR:

Recently, a polyurethane-type elastomer binder has been developed for use in solid propellants. The binder is synthesized by reaction of a liquid polyester with a diisocyanate.

BSPR:

According to structure and molecular weight, the known polyesters presently employed are viscous liquids, solids resins or wax-like products. Some of these known polyesters are hydroxy-terminated, having a molecular weight of about 2000, and are obtained through the polymerization of a mixture of .epsilon.-caprolactone and .epsilon.-methyl-.epsilon.-caprolactone in the presence of ethylene glycol or of glycerol. These polyesters have been used in the manufacture of polyurethane foam rubber.

BSPR:

At present, this type of hydroxy-terminated polyester, specifically polylactone polyols, are used in the synthesis of polyurethane elastomers. For example, the NIAx.RTM. polyols PCP0240 and PCP0300 are used as diols and triols, respectively, in elastomeric binding agents for solid propellants. However, PCP0240 has a wax-like consistency at ambient temperature, melts at temperature above 50.degree. C. and has a viscosity of 0.65 N.s/m.sup.2 at 55.degree. C.

BSPR:

PCP0300 is liquid at ambient temperature and has a viscosity of 22.5 N.s/m.sup.2 at 20.degree. C. The high viscosity of these products does not allow for the mixing of aluminized propellant compositions which contain more than 80% solids. Moreover, the glass transition temperature (Tg) of polyurethane elastomers obtained from these products is approximately -70.degree. C., i.e. lower than that of polyoxypropylene (-40.degree. C.) and almost as low as polybutadienes (-78.degree. C.).

BSPR:

According to yet another aspect of the invention, novel polyurethane binders useful in the production of solid propellants are contemplated, said binders being formed by reacting one of the aforementioned novel polyesters with a diisocyanate e.g. isophorone diisocyanate (IPDI).

BSPR:

According to yet another aspect of the invention solid propellants of high solids loading are contemplated comprising 12% w/w of a novel polyurethane binder as mentioned above and 88% w/w solids including ammonium perchlorate and aluminum.

DEPR:

A mixture of a diol selected from 1,2-propanediol (PG), 1,3-butanediol (13BD) and 3-methyl-1,5-pentanediol (3M15P) (1.1 mole), a dicarboxylic acid (adipic (AA) or sebacic (SA) 1.0 mole) and .epsilon.-caprolactone (1.0 mole) is heated to 200.degree.-250.degree. C. in nitrogen atmosphere in presence of a catalyst (TOGT, boron trifluoride). Volatile products are evaporated through vacuum

heating at 225.degree. C. The extent of the reaction is measured by the determination of the acid group and the quantity of non-polymerized .epsilon.-caprolactone. The hydroxyl concentration, the molecular mass are determined by a vapour phase osmometry and chromatography on a permeable gel, as well as the viscosity (see Table VIII).

DEPR:

We prepared elastomers from a mixture of Empol.RTM. co-polyester diol 1014BO/.epsilon.-CL and Empol.RTM. co-polyester triol 1040BO/.epsilon.-CL. For comparison purposes, an elastomer was made from R45M (trade mark for a hydroxy-terminated polybutadiene sold by Arco Chemical Corp.). These products were transformed into elastomeric polyurethane by reaction with a diisocyanate. The mixtures were poured into Teflon.RTM. moulds and placed into a 60.degree. C. oven to constant shore A hardness. The mechanical properties of the binders (.sigma..sub.m, .sigma..sub.r, .epsilon..sub.m, .epsilon..sub.r, and E) were measured at ambient temperature (23.degree. C.) using a Instron machine (Instron Canada Ltd., model TTC 1114) on rings manufactured with a punch. These rings had an interior diameter of 3.17 cm. Samples are drawn at a speed of 0.212 cm/s. The effective gauge length of the test tube was 3.50 cm. Mechanical properties are shown in Table IX. The polyester elastomers have an energy comparable to polybutadienes. Exemplified diisocyanate are DDI diisocyanate, a trade mark for an intermediate made from a 36-carbon dimer aliphatic dibasic acid; acid; isophorone diisocyanate (IPDI); and Isonate 901, a trade name for a polyfunctional isocyanate from Upjohn Polymer Chemicals of Kalamazoo, Mich.

DEPC:

Preparation of polyurethane binder from Empol co-polyester diol 1014BO/.epsilon.-CL, Empol co-polyester triol 1040BO/.epsilon.-CL and a diisocyanate

DEPC:

Preparation of polyurethane binders from a terpolymer diol, from polycaprolactone triol and from diisocyanate

DETL:

TABLE IX

Mechanical properties of polyurethanes from Empol copolyester diol and isocyanates Equivalent weight of R45M (lot 006121): 1420 g; heating time: to constant hardness; heating temperature: 60.degree. C.; isodecyl pelargonate: 25% of the elastomer; equivalent ratio diol/triol: 1.0 Test Diol Triol Diisocyanate .sigma..sub.m .epsilon..sub.m E Energy No. Name Name Name Mole MPa cm/cm MPa J

	.sigma..sub.m	.epsilon..sub.m	E	Energy	No.	Name	Name	Name	Mole	MPa	cm/cm	MPa	J
R45M DDI	1.0	0.223	3.72	0.13	0.51	46	Empol	1014BO/.epsilon.-CL	Empol				
1040BO/.epsilon.-CL IPDI	1.0	0.167	7.88	0.037	0.52	47	Empol	1014BO/.epsilon.-CL	Empol				
Empol 1040BO/.epsilon.-CL IPDI	1.25	0.321	2.50	0.209	0.41	48	PCP	0	240	PCP	0300		
IPDI	1.00	11.7	7.50	8.43	31.5	49	AGE-PTA-.epsilon.-CL	PCP	0	300	IPDI	1.00	0.404
1.35	0.344	0.21	44	PG-SA-.epsilon.-CL	I	SONATE901	0.80	8.20	66	.epsilon.-CL-PG-SA	"		
0.80	0.260	15.9	0.20	0.87	86	.epsilon.-CL-13BD-SA	"	0.088	4.8	0.393	0.17	117	
.epsilon.-CL-13BD-SA	"	1.20	0.330	3.7	0.19	0.59	147	.epsilon.-CL-BO-CHDA	"	1.20			
0.306	3.8	0.11	0.40										

DETL:

TABLE XI

Epoxides or diols or lactones R.sub.3

1,2-epoxy cyclohexene (ECH) yield 1,2-cyclohexene 4-vinyl-1, 2-epoxy cyclohexene " 4-vinyl-1, 2-cyclohexene Allyl-glycidyl ether (AGE) " 3-propenyloxy-1, 2-propylene Phenyl glycidyl ether (PGE) " 3-phenoxy-1, 2-propylene Polyesteramine diol " CH.sub.2 --CH.sub.2 --N(CH.sub.3)CH.sub.2 CH.sub.2 N--methyldiethanolamine " N--Methyldietheneamine .GAMMA.-butyrolactone " 1, 3-propylene .GAMMA.-valerolactone " 1, 3-butylene Propylene oxide " 1, 2-propylene Butylene oxide " 1, 2-butylene Propylene glycol " 1, 2-propylene 1,3-Butanediol " 1, 3-butylene 3-Methyl-1, 5-pentanediol " 3-methyl-1, 5-pentamethylene Niax polyol PCP 0530 " Polycaprolactone

Lactones, acids, anhydride R.sub.4

.delta.-valerolactone yield tetramethylene .GAMMA.-butyrolactone " 1, 3-propylene .GAMMA.-valerolactone " 1, 3-butylene cis-1,3-cyclohexane dicarboxylic " 1, 2-cyclohexene anhydride (CHDA) Methyl succinic anhydride (MSA) " 1, 2-propylene Methyl succinic acid " 1, 2-propylene 3-methyl glutaric anhydride " 2-methyl-1,3-propylene (MGA) or acid Phthallic anhydride (PA) or acid " Phenylene Adipic acid (AA) tetramethylene Sebacic acid (SA) octamethylene (CH.sub.2).sub.8 Empol 1014 C.sub.36 radical

CLPR:

1. A process for the preparation of a polyurethane binder comprising reacting a diisocyanate with a block or sequenced co-polyester diol of the structural formula I

CLPR:

5. A polyurethane binder for use in a solid propellant comprising the reaction product of a diisocyanate and a block or sequenced co-polyester diol produced by the process of claim 1 and having the structural formula

CLPR:

6. The polyurethane binder according to claim 5 in which the diisocyanate is selected from the group consisting of isophorone, diisocyanate, and intermediate made from a 36-carbon dimer aliphatic dibasic acid, and a polyfunctional isocyanate.

CLPR:

7. The polyurethane binder composition according to claim 5 or 6 further including a plasticizer.

CLPR:

8. A polyurethane binder according to claim 7 in which the plasticizer is isodecyl pelargonate.

CCOR:

149/19.4

CCXR:

149/114

CCXR:

149/76